

# INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> :		(11) International Publication Number:	WO 97/38302
G01N 27/64	A1	(43) International Publication Date:	16 October 1997 (16.10.97)

(21) International Application Number: PCT/US97/05641

(22) International Filing Date: 4 April 1997 (04.04.97)

(30) Priority Data:

08/627,584

08/628,147

08/726,843

4 April 1996 (04.04.96)

US

4 April 1996 (04.04.96)

US

08/726,843

8 October 1996 (08.10.96)

US

(71) Applicant: MINE SAFETY APPLIANCES COMPANY [US/US]; RIDC Industrial Park, 121 Gamma Drive, Pittsburgh, PA 15238 (US).

(72) Inventors: PERTINARIDES, John, M., A.; 2814 Kinloch Drive, Orlando, FL 32817 (US). TARASSOV, Alexandre, S.; 135 Woodhaven Drive, Mars, PA 16046 (US). CARNAHAN, Byron, L.; 9783 Brant Avenue, Pittsburgh, PA 15237 (US). PIPICH, Charles, W.; 212 Rush Valley Road, Monroeville, PA 15146 (US). KOUZNETSOV, Viktor; 132 Woodhaven Drive, Mars, PA 16046 (US). UBER, Robert, E.; 4137 North Ampton, Allison Park, PA 15101 (US).

(74) Agent: WETTACH, Thomas, C.; Titus & McConomy, 20th floor, Four Gateway Center, Pittsburgh, PA 15222 (US).

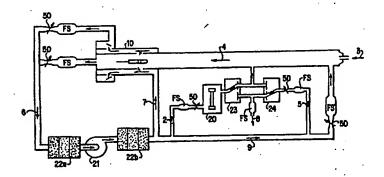
(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, IP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG). Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

#### Published

With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: RECIRCULATING FILTRATION SYSTEM FOR USE WITH A TRANSPORTABLE ION MOBILITY SPECTROMETER



#### (57) Abstract

Generally, the present invention provides a recirculating filtration system for use with a transportable ion mobility spectrometer. A first preferred embodiment of the transportable recirculating filtration system comprises a pump connected with a set of filters and flow sensors to an IMS sensor. The IMS sensor's outlet flow is cleaned by the filters and recirculated by the pump back into the IMS sensor as the carrier fluid stream flow. A portion of the IMS sensor's outlet flow equal to the amount of flow introduced into the sensor is exhausted from the filtration system to maintain a constant total flow volume through the system as the sample is being taken. A second preferred embodiment of the present invention provides a transportable recirculating filtration system for measuring an unknown concentration of a selected ionizable contaminant substance such as benzene present in which other substances may coexist, which employs a device for introducing a controlled concentration of the selected ionizable contaminant into an ion mobility spectrometry (IMS) sensor. The unknown concentration is calculated using the Standard Addition Method. The second preferred embodiment of the present invention can also be used to calibrate the IMS sensor by establishing which ionogram structures result from the presence of a particular analyte in the sample stream. A third preferred embodiment of the present invention provides a recirculating filtration system for use with a transportable ion mobility spectrometer in gas chromatography applications. The transportable recirculating filtration system comprises a pump and a set of filters and flow sensors connected to an ion mobility spectrometry sensor having a gas chromatograph column at its inlet.

# FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL Albania AM Armenia AT Anstria AU Australia AZ Azerbaijan BA Boania and Herzegovina BB Barbados BB Belgium BBF Burkina Paso BG Bulgaria BJ Benin BR Brazil BY Belarus CA Canada Crentral African Republic CG Congo CH Switzerland COte d'Ivoire CM Cameroon CM China CU Caba CZ Czech Republic CE Cermany KE Demmark E Batonia	ES Spain FI Finland FR Prance GA Gabon GB United Kingdom GE United Kingdom GE Georgia GH Ghana GN Guinea GR Greece HU Hungary IE beland IL krael IS leeland FT kaly JP Japan KE Kenya KG Kyngyzstan KP Democratic People's Republic of Korea KR Republic of Korea KR Republic of Korea KZ Kazakatan LC Saint Lucia LL Liechtenstein LK Sri Lenka LR Liberia	LS Lesotho LT Lithuania LU Luxembourg LV Latvia MC Monsco MD Republic of Moldova MG Madagascar MK The former Yugoslav Republic of Macedonia ML Mail MN Mongolia MR Mauritania MW Malawi MX Mexico NE Niger NL Netheriands NO Norway NZ New Zealand PL Poland PT Portugal RO Romania RU Russian Federation Sudan SE Sweden SG Singapore	SI Slovenia SK Slovakia SN Senegal SZ Swaziland TD Chad TG Togo TJ Tajikistan TM Turkmenistan TR Turkey TT Trinidad and Tobago UA Ukraine UG Uganda US United States of America UZ Uzbekistan VN Viet Nam YU Yagoslavia ZW Zimbahwe

### TITLE

# RECIRCULATING FILTRATION SYSTEM FOR USE WITH A TRANSPORTABLE ION MOBILITY SPECTROMETER

### FIELD OF THE INVENTION

The invention relates generally to a transportable recirculating filtration system for use in conducting measurements with an ion mobility spectrometer (IMS), and also to a transportable recirculating filtration system for using a field IMS sensor in gas chromatography applications.

## BACKGROUND OF THE INVENTION

U.S. Patent No. 5,420,424 provides a sensor using ion mobility spectrometry (IMS) to detect trace concentration level species present in a sample gas stream. The IMS sensor disclosed in U.S. Patent No. 5,420,424 utilizes periodic electrical fields to separate different species of ions according to the functional dependence of their mobility with electric field strength. Ions generated in the ionization chamber of the IMS sensor are guided through an ion filter to an ion detector by an electric field known as the "dispersion field." This "dispersion field" is created by an asymmetric periodic radio frequency (RF) voltage applied between a pair of closely spaced longitudinal electrodes. The displacement of the ions induced by the dispersion field is modified or compensated by a second electric field known as the "compensation field." The compensation field is created by an adjustable time independent direct current electrical potential that is applied between the electrodes to isolate a particular ion species for detection as a result of the variance in mobility between particular ion species as a function of electric field strength. This form of ion mobility spectrometry, known as field ion spectrometry, offers a new method of detecting species present at trace (parts per million to parts per trillion) concentration levels in a sample gas to be analyzed.

To avoid compromising the sensor's performance due to impurities which may be present in the carrier fluid stream that transports the ions through the ion filter, existing IMS sensor designs require connection to a filtration system to clean the carrier fluid stream flow prior to its introduction into the sensor. The use of a stationary filtration source presents a problem if the IMS sensor is to be used as a portable instrument in the detection of atmospheric contaminants in remote locations. Existing portable filtration systems used with IMS devices are not compatible with the IMS design disclosed in U.S. Patent No. 5,420,424. The present invention solves this problem by providing a recirculating filtration system for use with a transportable IMS sensor of the design disclosed in U.S. Patent No. 5,420,424.

One preferred embodiment of the present invention provides a recirculating filtration system for use with a transportable IMS sensor in measuring an unknown concentration of a selected ionizable contaminant substance present in a sample in which other substances may coexist. The ambient air sample containing the contaminant of interest can compromise IMS performance due to the variable amount of humidity and trace species present in the ambient air stream. The accuracy of the sensor's concentration measurement can be adversely affected by charge transfer reactions which occur between the ionizable contaminant of interest and other ionizable species present in the ambient air sample. Charge transfer reactions can either enhance or diminish the net efficiency with which the contaminant of interest is ionized. Consequently, the sensor's apparent response would depend not only on the concentration of the contaminant of interest in the ambient air sample, but also on the concentration of other substances which contribute to its ionization through these charge transfer reactions. The adverse effects of charge transfer reactions on the IMS sensor's performance can be eliminated through the use of a

filtration system configuration which introduces a known concentration of the ionizable contaminant of interest into a fluid stream containing an unknown concentration of the selected contaminant. This permits utilization of a measurement technique known as the Standard Addition Method to quantify the unknown concentration. The Standard Addition Method compares the IMS measurement of the unknown concentration level to the known concentration level in a manner that eliminates the effects of charge transfer reactions on the result. This embodiment of the present invention can also be used to calibrate the IMS sensor by establishing which ionogram structures result from the presence of a particular analyte in the sample stream. A known concentration of an analyte is introduced into the sample stream. The resulting ionogram is compared to an ionogram recorded with no contaminants present. By comparing these spectra, a correlation can be made between the positions of features in the ionogram and the analyte which gives rise to these features.

Another preferred embodiment of the present invention provides a recirculating filtration system for use with a transportable IMS sensor of the design disclosed in U.S. Patent No. 5,420,424 that is configured for use in gas chromatography applications. That embodiment also provides a heating device for significantly minimizing the effects of adsorption taking place on the IMS sample stream inlet surface. This decreases the IMS sensor's response time and recovery time characteristics with regard to detecting rapidly changing analyte concentration levels. The device provides heating to the IMS sensor's sample stream inlet to increase the temperature of the sample stream inlet surface. This increased temperature results in less time between adsorption and desorption of the analyte on the sample stream inlet surface, which causes a greater portion of the overall analyte concentration level to be detected by the IMS sensor. This results in a significant reduction in the time necessary for the analyte to reach equilibrium density on the sample

stream inlet surface, thus decreasing the IMS sensor's response time and recovery time characteristics with regard to detecting rapidly changing concentration levels. The heating device may be optionally configured with a flow smoothing insert for reducing the turbulence in the fluid stream flow entering the IMS sensor's carrier stream plenum inlet. Elimination of this turbulence is necessary in order to prevent mixing of the carrier and sample fluid streams in the ion filter, which would otherwise adversely affect the IMS sensor's sensitivity due to the impurities present in the sample fluid stream. Without the use of a flow smoothing insert, a reduction of the carrier stream flow is necessary to eliminate this turbulence, which in turn adversely affects the IMS sensor's sensitivity. This decreased sensitivity is caused by the increased amount of time required for the ionized substance to traverse the ion filter. Extending the time interval which ions spend in the ion filter allows more time for loss mechanisms (such as diffusion to the filter's walls and charge neutralization reactions) to attenuate the ion current which reaches the ion detector. The flow smoothing insert thus reduces turbulence in the carrier stream flow, allowing a return of the flow to a level that restores the IMS sensor's sensitivity.

# SUMMARY OF THE INVENTION

Generally, the present invention provides a recirculating filtration system for use with a transportable ion mobility spectrometer. A first preferred embodiment of the transportable recirculating filtration system comprises a pump connected with a set of filters and flow sensors to an ion mobility spectrometry sensor preferably of the design disclosed in U.S. Patent 5,420,424. The IMS sensor's outlet flow is cleaned by the filters and recirculated by the pump back into the IMS sensor as the carrier fluid stream flow. A portion of the IMS sensor's outlet flow equal to the amount of flow introduced

into the sensor is exhausted from the filtration system to maintain a constant total flow volume through the system as the sample is being taken.

A second preferred embodiment of the present invention provides a transportable recirculating filtration system for measuring an unknown concentration of a selected ionizable contaminant substance such as benzene present in a sample fluid stream in which other substances may coexist. The filtration system of this alternate preferred embodiment comprises a pump connected with a set of filters, flow sensors, and a plurality of valves to a device for introducing a controlled concentration of the selected ionizable contaminant into an ion mobility spectrometry sensor preferably of the design disclosed in U.S. Patent 5,420,424. The measurement of an unknown concentration of the selected contaminant preferably comprises the following steps: (1) combining the controlled contaminant concentration with an unknown concentration of the same contaminant to measure the IMS sensor's response to the combined controlled and unknown concentrations; (2) measuring the IMS sensor's response to only the unknown concentration; and (3) determining this unknown concentration by comparing the measurement in step (1) to the measurement in step (2). This embodiment of the present invention also provides a recirculating filtration system which can be used to calibrate the IMS sensor by establishing which ionogram structures result from the presence of a particular analyte in the sample stream. A known concentration of an analyte is introduced into the sample stream. The resulting ionogram is compared to an ionogram recorded with no contaminants present. By comparing these spectra, a correlation can be made between the positions of features in the ionogram and the analyte which gives rise to these features.

A third preferred embodiment of the present invention provides a recirculating filtration system for use with a transportable IMS sensor of the design disclosed in U.S. Patent No. 5,420,424 that is configured for use in gas chromatography applications. The gas chromatograph is preferably comprised of a multicapillary column containing a coating comprised of a stationary phase substance. The multicapillary column is in communication with the IMS sensor sample stream inlet for transmitting an unknown concentration level of at least one analyte into the IMS sample stream inlet to be measured by the ion mobility spectrometer. A source of carrier gas in communication with a sample injector entrains the analyte in the sample injector for transport into the multicapillary column. The analyte is soluble in the stationary phase substance such that transport of the analyte into the IMS sample stream inlet is delayed by a predetermined "retention time" interval in the multicapillary column. Variation in the retention time between different analyte species allows the IMS sensor to accurately measure the individual concentrations of the various analyte species since the entry of each analyte species into the IMS sample stream inlet will be staggered as compared to the other species present in the sample stream.

Preferably, the multicapillary column is sealed to a heated transfer line leading to the sample stream inlet of the IMS sensor. The transfer line is preferably maintained at a temperature 10°C - 40°C above that of the multicapillary column in order to ensure that analyte species will not adsorb on the walls of the transfer line instead of entering the sample stream inlet of the IMS sensor. The transfer line can be optionally configured with a flow smoothing insert to produce a laminar flow profile throughout the volume between the transfer line outlet end and the IMS sensor housing upon exit of the IMS carrier flow stream from the flow smoothing insert. The heating provided by the transfer

line also increases the temperature of the sample stream inlet surface to reduce the amount of time between adsorption and desorption taking place on the surface. This significantly improves the ability of the IMS sensor to follow rapidly changing analyte concentration levels. The inlet heating device comprises a hollow metal passageway for passage of a sample fluid stream from the outside environment into the sample stream inlet of the ion mobility spectrometer. A hollow metal housing is preferably hermetically welded to both the passageway inlet and outlet ends for mounting the device to the sample stream inlet of the ion mobility spectrometer using a ceramic flange. A heating element preferably comprised of a high resistance wire surrounds the surface of the passageway for electrically heating the passageway. A temperature control circuit controls electrical power input to the heating element to maintain the passageway at a substantially constant reference value. The temperature control circuit comprises at least one temperature measuring device, preferably an electrical resistance-measuring thermometer, attached to the surface of the passageway for measuring the temperature of the passageway. A temperature controller having an input electrically connected to the temperature measuring device and an output electrically connected to the heating element compares the temperature measured by the temperature measuring device to a predetermined reference value and adjusts the electrical power input to the heating element to substantially match the temperature of the passageway to the reference value.

The heated transfer line is optionally provided with a flow smoothing device for decreasing the turbulence present in the fluid flow entering the IMS sensor's carrier stream inlet. This flow smoothing insert permits increasing the fluid flow rate entering the IMS sensor's carrier stream inlet to levels which maximize the IMS sensor's measurement sensitivity without causing mixing of the sample and carrier fluid stream

flows. The flow smoothing element comprises an insert of porous material, preferably a disc- shaped metal mesh with a center opening that fits over the housing of the heating device, which is placed between the heating device housing and the surface of the sample stream inlet at a location proximate to the outlet end of the heating device. The flow smoothing insert fills the gap between the heating device housing and the sample stream inlet plenum surface, causing the velocity of the fluid stream flow entering the carrier stream inlet to be equalized across the outlet side of the flow smoothing element to eliminate flow turbulence. Preferably, the flow smoothing insert is electrically isolated from the IMS sample stream inlet plenum surface by a band of electrically insulating material placed between the insert and the IMS sample stream inlet surface.

Other details, objects, and advantages of the present invention will become apparent in the following description of the presently preferred embodiments.

# BRIEF DESCRIPTION OF THE DETAILED DRAWINGS

In the accompanying drawings, the preferred embodiments of the present invention and preferred methods of practicing the present invention are illustrated wherein:

Figure 1 is a schematic fluid system diagram of a first preferred embodiment of the present invention which provides a recirculating filtration system for use with a transportable IMS sensor.

Figure 2 is a schematic fluid system diagram of a second preferred embodiment of the present invention providing a portable recirculating filtration system for measuring an unknown concentration of a selected ionizable contaminant substance when present in a sample fluid stream in which other substances may coexist.

Figure 3 is a schematic fluid system diagram of a third preferred embodiment of the present invention which provides a recirculating filtration system for use with a transportable IMS sensor in gas chromatography applications.

Figure 4 is a cross-sectional view of a preferred heating device for the present invention.

Figure 5 is a cross-sectional view of an alternate embodiment of a heating device as configured with a flow smoothing insert.

Figure 6 is a simplified representation of an IMS sensor's response time and recovery time characteristic in detecting a stepwise increase in the concentration of the analyte with and without heating of the ionization chamber inlet.

Figure 7 is a graph of the temperature profile across a preferred heating device.

Figure 8 is a graph illustrating the effect of the use of a preferred inlet heating device on an IMS sensor's response time and recovery time characteristic to changes in analyte concentration levels.

Figure 9A shows a typical set of data collected by an IMS sensor without a flow smoothing insert attached to the heating device; and

Figure 9B shows the increased sensitivity of an IMS measurements with a flow smoothing insert attached to the heating device.

### DETAILED DESCRIPTION OF THE PRESENTLY PREFERRED EMBODIMENTS

Figure 1 shows a first preferred embodiment of the present invention, which provides a recirculating filtration system for use with a transportable IMS sensor. The embodiment shown in Figure 1 comprises a pump 21 in combination with a set of filters 22a and 22b in the inlet and outlet streams thereof, respectively, along with a plurality of flow sensors FS and flow adjustment valves 50 all in communication with an ion mobility

spectrometry (IMS) sensor 10. IMS sensor 10 is preferably of the design described in U.S. Patent 5,420,424, the disclosure of which is incorporated herein by reference. The IMS sensor's outlet fluid flow stream 6 is purified and recirculated back into the IMS sensor 10 as the carrier fluid stream flow 7 described in U.S. Patent 5,420,424. A portion of the IMS sensor's outlet fluid flow stream 6 equal to the amount of sample fluid stream flow 3 introduced into the IMS sensor 10 is removed as exhaust 8 from the filtration system, thereby maintaining a constant total flow rate through the system as the sample is being taken. The flow rates of the sample 3 and exhaust 8 fluid streams are typically in the range of 10 to 500 milliliters per minute, while typical carrier fluid stream 7 flow rates are in the range of 2 to 4 liters per minute.

Pump 21, preferably of the ASF Model No. 5010 or 7010 Oil-less Diaphragm

Pump design, circulates fluid stream flow through the system. Filters 22a and 22b,

preferably consisting of 100 to 500 cubic centimeters (cc) of activated charcoal combined

with 200cc to 1000cc of combined type 5A and 13X molecular sieve, clean the outlet

fluid flow stream 6 prior to recirculation back into the IMS sensor 10 as the carrier gas 7

described in U.S. Patent 5,420,424. A plurality of flow sensors FS, preferably

Honeywell Model AWM3000 or AWM5000 series mass flow sensors, are installed to

measure flow rates at various points throughout the system. A plurality of flow

adjustment valves 50, preferably Model No. SS-2MG4 valves manufactured by Newpro,

Inc., are adjusted to initially set the various flow rates in the system to their proper

values. These valves 50 are then left at their initial positions during use of the system in

making IMS measurements.

The recirculating filtration system displayed in Figure 1 will permit operation of a portable IMS sensor for the detection of selected atmospheric contaminants in remote locations. However, the accuracy of the sensor's concentration measurement can be adversely affected by charge transfer reactions which occur between the ionizable contaminant of interest and other ionizable species present in the ambient air sample. Figure 2 displays a second preferred embodiment of the present invention, which eliminates the effect of charge transfer reactions on the selected ionizable contaminant substance to permit accurate measurement of an unknown concentration level Cs of the selected contaminant in a sample fluid stream flow 3 in which other substances may coexist.

The recirculating filtration system of Figure 2 is also preferably designed for use with the IMS sensor 10 disclosed in U.S. Patent No. 5,420,424. The filtration system of Figure 2 preferably comprises a permeation calibration device 20, a pump 21, a set of filters 22a and 22b, flow sensors FS, and two three-port two-way solenoid valves 23 and 24 with interconnecting fluid flow passageways and flow adjustment valves 50. Preferably, a conventional permeation calibration device 20, such as that disclosed in U.S. Patent Nos. 4,399,942 or 4,715,217, introduces a controlled concentration Cpd of the selected ionizable contaminant substance, by way of fluid stream 2, into the total flow 4 entering the sample stream inlet of the IMS sensor. Fluid stream 2 passes through permeation calibration device 20 to permit calibration of the IMS sensor 10 response to that expected for the ion concentration introduced, and to permit use of the Standard Addition Method in calculating an unknown concentration of the selected contaminant as measured by the IMS sensor 10. Other means of introducing a controlled concentration Cpd of the selected ionizable contaminant into the system that can be used are diffusion

vials, diffusion tubes, standards from pressurized calibration cylinders, and other methods such as those disclosed in <u>Gas Mixtures</u>: <u>Preparation and Control</u>, by Gary O. Nelson (1992).

Preferably, the permeated fluid making up the controlled concentration Cpd consists of a substance such as benzene, contained in the permeation calibration device 20 in liquid form and released into the calibration fluid stream 2 in gaseous form, but the filtration system design shown in Figure 2 can also be adapted for the use of volatile solids or volatile liquids having a boiling point between 40C and 125C, as well as other single phase gases, two-phase solid gas systems or two-phase liquid gas systems desired for use as the permeated fluid. In addition to the permeated fluid, the calibration stream 2 preferably consists of an inert gas or an inert mixture of gases such as that found in dehumidified air which has been filtered through activated charcoal. Typical permeated fluid emission concentrations are between 0.1 and 10 parts per million for calibration fluid stream 2 flow rates in the range of 50 to 200 milliliters per minute.

Pump 21, preferably of the ASF Model No. 5010 or 7010 Oil-less Diaphragm

Pump design, circulates flow through the system. Filters 22a and 22b, which preferably

consist of 100 to 500 cubic centimeters (cc) of activated charcoal combined with 200 to

1000 cc of combined type 5A and 13X molecular sieve, clean the outlet fluid stream flow

6 from the IMS sensor 10 prior to recirculation back into the IMS sensor 10 as the carrier

fluid stream 7 described in U.S. Patent 5,420,424. A first portion of the cleaned outlet

flow 6 not introduced into the carrier fluid stream 7 is circulated through the permeation

calibration device 20 as the calibration stream 2 and then into the total flow 4 entering the

sample stream inlet of the IMS sensor 10. A second portion of the cleaned outlet flow 6

not introduced into the carrier fluid stream 7 is recirculated back into the total flow 4

entering the sample stream inlet of the IMS sensor 10 as the makeup component 5 of the recirculated dilution flow 9 that dilutes the sample 3 to the level necessary for measurement of the unknown concentration. A third portion of the cleaned outlet flow 6 not introduced into the carrier stream 7 is removed as exhaust 8 from the filtration system to maintain a constant total flow volume through the system as the sample is being taken. This exhaust flow 8 is equal to the amount of sample fluid stream flow 3 introduced into the IMS sensor 10. Sample 3 and exhaust 8 fluid stream flow rates delivered by the system are typically in the range of 10 to 500 milliliters per minute, while typical carrier fluid stream 7 flow rates are in the range of 2 to 4 liters per minute.

A plurality of flow sensors FS, preferably Honeywell Model AWM3000 or AWM5000 series mass flow sensors, are installed to measure flow at various points throughout the system. A plurality of flow adjustment valves 50 are adjusted to initially set the various flow rates in the system to their proper values. These valves 50 are then left at their initial positions during use of the system in making IMS measurements. Valve 23, preferably of the design type LHDA1211111H manufactured by the Lee Company, directs the calibration fluid stream 2 from the permeation calibration device 20 into the total flow 4 entering the sample stream inlet of the IMS sensor 10, or out of the system as required. Valve 24, again preferably of the Lee LHDA1211111H design type, adjusts the makeup component 5 of the recirculated dilution flow 9 such that a constant total IMS sample stream inlet flow 4 is maintained for comparing the controlled concentration of the selected contaminant present in the calibration flow 2 to the unknown concentration to be measured in the sample 3. This constant total IMS sample stream inlet flow 4 permits use of the Standard Addition Method in calculating the unknown concentration Cs from the known controlled concentration Cpd. A conventional IMS sensor 10 will work with the

system, but the preferred embodiment is designed for use with the IMS sensor disclosed in U.S. Patent No. 5,420,424.

The measurement of an unknown concentration of the selected contaminant preferably comprises the following steps: (1) combining the controlled contaminant concentration with an unknown concentration of the same contaminant to measure the IMS sensor's response to the combined controlled and unknown concentrations; (2) measuring the IMS sensor's response to only the unknown concentration; and (3) determining this unknown concentration by comparing the measurement in step (1) to the measurement in step (2).

In the first step, valve 23 is positioned to introduce controlled concentration Cpd into the total flow 4 entering the IMS sample stream inlet. Valve 24 is positioned to divert makeup flow 5 out of the system as the exhaust 8. The positioning of valve 24 permits the sample 3 to be introduced into the total flow 4 entering the IMS sample stream inlet. The positioning of valve 24 in concert with the pre-adjustment of the valves 50 causes the sample flow 3 to equal 10% of the total flow 4 entering the IMS sample stream inlet. This 10% dilution factor is necessary to dilute the unknown concentration of the selected contaminant in the sample 3 to a level that will ensure a linear response of the IMS sensor 10 to changes in the unknown concentration level, but the flow rates preset by the valves 50 could be adjusted to provide for other dilution factors. The IMS sensor 10 measures the combined signal output Stotal produced by ionization of both the controlled concentration Cpd and the unknown concentration Cs of the selected contaminant. Stotal can be characterized by the equation Stotal = Spd + Ss = A\*Cpd + 0.1\*A\*Cs. A is a constant of proportionality between the electrical signal output S of the IMS 10 and the contaminant concentration C and is generally unknown due to its

dependence on charge transfer reactions. The signal output Ss produced by the unknown concentration Cs reflects the 10% dilution factor created by valve 24.

In the second step, valve 23 is repositioned to direct calibration flow 2 out of the system. Valve 24 is repositioned to reinstate maximum makeup flow 5 in combination with the sample 3 as the total flow 4 entering the IMS sample stream inlet. The amount of increased makeup flow 5 provided by valve 24 compensates for the amount of calibration flow 2 redirected out of the system to keep the sample flow 3 equal to 10% of the total flow 4 entering the IMS sample stream inlet. The IMS sensor 10 measures the signal output Ss produced by ionization of only the unknown concentration Cs of the selected contaminant. Ss can again be characterized by the equation Ss = 0.1\*A\*Cs as described above.

After performance of the above described measurement sequence, the unknown concentration Cs of the selected contaminant present in the sample 3 is calculated by the Standard Addition Method. Since the controlled concentration Cpd is known, the unknown contaminant concentration Cs can be readily obtained by comparing signal output Ss to the combined signal output Stotal. This unknown concentration Cs is calculated by solving the equation Cs = 10\*Cpd/(R-1) where R = Stotal/Ss. Use of the Standard Addition Method to calculate the unknown concentration Cs corrects for the effects of charge transfer reactions on the IMS concentration measurement by eliminating the necessity of arriving at a value for generally unknown proportionality constant A. This calculation can be performed by means of an algorithm run on a computer processor by computer software programmed for this purpose that is stored on a computer-readable storage medium. Other means of calculating the unknown contaminant concentration Cs of the selected contaminant are also possible, such as use of an electronic integrated

differential amplifier circuit, an analog or a combined analog and digital comparison circuit comprised of discrete electrical and digital logic components, or other similar circuitry presently in the state of the art.

The second preferred embodiment of the present invention shown in Figure 2 also provides a recirculating filtration system which can be used to calibrate the IMS sensor 10 by establishing which ionogram structures result from the presence of a particular analyte in the sample stream. A known concentration of an analyte is introduced into the sample stream 3. The resulting ionogram is compared to an ionogram recorded with no contaminants present. By comparing these spectra, a correlation can be made between the positions of features in the ionogram and the analyte which gives rise to these features. Calibration of the IMS sensor 10 is accomplished by introducing only the controlled concentration Cpd of the selected contaminant into the IMS. This allows correlating the compensation voltage to generate an ionogram from the measurements taken by the IMS sensor 10. In the calibration step, valve 23 is positioned to introduce controlled concentration Cpd present in the calibration fluid stream 2 into the total flow 4 entering the IMS sample stream inlet. Valves 23 and 24 are positioned to provide maximum makeup flow 5. The lack of exhaust flow 8 out of the system prevents the entry of sample flow 3 into the IMS 10. The total IMS sample stream inlet flow 4 is passed through the IMS sensor 10 to calibrate the IMS sensor 10 output Spd. Spd can be characterized by the equation Spd = A'\*Cpd where A' is a constant of proportionality similar to that described above but which has a different value due to a different composition of the total IMS sample stream inlet flow 4. Use of this equation allows comparison of Spd to the output anticipated from ionization of only the controlled concentration Cpd of the selected contaminant. The resulting ionogram is compared to

an ionogram recorded with no contaminants present. By comparing these spectra, a correlation can be made between the positions of features in the ionogram and the analyte which gives rise to these features.

Figure 3 shows a third preferred embodiment of the present invention which provides a recirculating filtration system for use with a transportable IMS sensor in gas chromatography applications. The third preferred embodiment shown in Figure 3 again comprises a pump 21 in combination with filters 22a and 22b in the inlet and outlet streams thereof, respectively, along with a plurality of flow sensors FS and flow adjustment valves 50 all in communication with an IMS sensor 10. IMS sensor 10 is again preferably of the design described in U.S. Patent 5,420,424. The IMS sensor's outlet fluid flow stream 6 is again purified and recirculated back into the IMS sensor 10 as the carrier fluid stream flow 7 described in U.S. Patent 5,420,424. A portion of the outlet fluid flow stream 6 again equal to the amount of sample fluid stream flow 3 introduced into the IMS sensor 10 is removed as exhaust 8 from the filtration system, thereby maintaining a constant total flow rate through the system. The flow rates of the sample 3 and exhaust 8 fluid streams are typically in the range of 10 to 100 milliliters per minute, while typical carrier fluid stream 7 flow rates are in the range of 2 to 4 liters per minute.

Pump 21, again preferably of the ASF Model No. 5010 or 7010 Oil-less

Diaphragm Pump design, circulates fluid stream flow through the system. Filters 22a and

22b, again preferably consisting of 100cc to 500cc of activated charcoal combined with

200cc to 1000cc of combined type 5A and 13X molecular sieve, clean the outlet fluid

flow stream 6 prior to recirculation back into the IMS sensor 10. A plurality of flow

sensors FS, preferably of the Honeywell Model AWM3000 design, are installed to

measure flow rates at various points throughout the system. A plurality of flow adjustment valves 50, preferably Model No. SS-2MG4 valves manufactured by Newpro, Inc., are adjusted to initially set the various flow rates in the system to their proper values. These valves 50 are then left at their initial positions during use of the system in making IMS measurements.

As shown in Figure 3, sample fluid stream flow 3 originates from a source of high pressure carrier gas 70 and is injected through an injection solenoid valve 71 into a gas chromatograph preferably comprised of a multicapillary column 72. Carrier gas 70 is preferably injected into the multicapillary column 72 by way of a sample injector 73 prior to entering the sample stream inlet of the IMS sensor 10. Due to the preferred design of the system, the sample fluid stream flow rate 3 through the multicapillary column 72 will be equal to the exhaust fluid stream flow rate 8 and is controlled by means of an adjustable orifice flow restrictor valve 51 located in series with valve 71 and preferably manufactured by the Sibertech Company, Novosibirsk, Russia. Valve 71, which is preferably of the two-way three-port injection solenoid design manufactured by the Sibertech Company either: (i) is positioned to bypass sample injector 73 to allow pure carrier gas 70 to enter the IMS sensor 10 as the sample fluid stream flow 3, or alternately (ii) is positioned to allow the carrier gas 70 to pass through the sample injector 73 to entrain the analyte to be processed by the IMS sensor 10. Although a multicapillary column design is used as the gas chromatograph in the preferred embodiment, other single capillary and packed column designs in the current state of the art are acceptable for use, so long as a means of flow and temperature control are provided similar to that described herein, and so long as the gas chromatograph is hermetically sealed to the sample stream inlet of the IMS sensor.

The carrier gas 70 is introduced into the flow system at high pressure by means of a conventional pressure regulator and relief valve arrangement. The preferred embodiment utilizes a pressure regulator and relief valve system manufactured by Sibertech Company. Pressure in tank 69 is reduced by the pressure regulator 68 from 100 to 2500 psia at the outlet of tank 69 to 15 to 25 psig at head of the multicapillary column 72. The carrier gas 70 is preferably inert such as pure nitrogen or it can be ultrapure air. The disadvantage of using nitrogen as the carrier gas 70 is that when the instrument is first turned on some air will already be present in the IMS sensor 10 flow system. As the instrument operates, the pure nitrogen carrier gas will mix with and gradually displace this air. During this period, the composition of the recirculating gas in the IMS sensor 10 flow system will gradually shift from air to nitrogen. However, stable readings cannot be achieved with the IMS sensor 10 until the composition of the recirculating gas for the IMS sensor 10 is stable. Thus operation with nitrogen as the carrier gas 70 would extend the time required for the IMS sensor 10 to reach stable operation. Ultrapure air is the source of carrier gas 70 used in the preferred embodiment.

The sample injector 73 is a hollow container preferably made of metal such as stainless steel and having one wall which is hermetically sealed to the multicapillary column 72. In one configuration, a second wall of the sample injector 73 is comprised of a septum membrane. This membrane is preferably made of silicon or other similar materials such as a Teflon-type elastomer. A gas or liquid sample to be processed by the IMS sensor 10 is preferably injected through the membrane by means of syringe. Since the sample injector 73 is at an elevated temperature (100°C or greater), liquid samples quickly vaporize once they enter the sample injector 73 volume. In an alternate arrangement, the membrane wall of the sample injector is removed and a sampling

cartridge is inserted into the injector. Once in the sample injector 73 the cartridge is rapidly heated, desorbing materials which have been collected on the cartridge's wire mesh collecting surface. In either configuration, the vapor samples present in sample injector 73 are forced onto the multicapillary column 72 by momentarily supplying a pulse of carrier gas 70 to the sample injector 73 volume. The sample fluid stream flow 3 entering the sample stream inlet of the IMS sensor 10 is thus a mixture of carrier gas 70 entrained with unknown concentrations of one or multiple analytes to be analyzed. Sample fluid stream flow 3 exiting the sample injector 73 is preferably introduced at a rate of 10-100 ml/min and preferably between 50 and 80 ml/min into the multicapillary column 72. The multicapillary column 72 is preferably a hollow bundle of glass capillary tubes that contain a lining known as the stationary phase. These glass tubes range from approximately 25 to 100 micrometers (um) in inner diameter (ID) and are preferably approximately 40 um in ID. The stationary phase can be comprised of substances such as SE-30, SE-54 or carbowax 20M, with SE-54 being used in the preferred embodiment.

In operation of the system, the carrier gas 70 first passes through the sample injector 73 where samples of the analyte are injected in a pulse-like manner into the carrier gas 70 thereby giving rise to spatially separated carrier gas "slugs" containing various species of the entrained analyte. These entrained analyte carrier gas slugs are then passed through the multicapillary column 72 at the carrier gas 70 flow rate. The molecules comprising the various analyte species present in the sample injector 73 are each soluble to differing extent in the stationary phase substance. Thus all the analyte species entrained in a given carrier gas slug will undergo numerous passages in and out of solution with the stationary phase substance as the carrier gas slug transits the length of the multicapillary column 72. Each analyte species of interest will experience a transit

time through the multicapillary column 72 different from the other analyte species present in that carrier gas slug due to the differing extent to which each analyte species is soluble in the stationary phase. This transit time is known as the "retention time" for that particular analyte species. This variation in retention times allows the IMS sensor to achieve improved selectivity in distinguishing between the various analyte species entrained in the carrier gas 70 since the entry of each analyte species into the sample stream inlet of the IMS sensor 10 will be staggered as compared to the other species present in the carrier gas 70. Thus the IMS sensor 10 is required to respond to only a single analyte species at a time thereby preventing a degradation in the IMS measurement by the other species present in the analyte sample.

The multicapillary column 72 is heated by a surrounding oven and maintained within a selected temperature falling within the band of 100-180°C to ensure the optimum retention time distribution for the various analyte species of interest. The multicapillary column 72 is hermetically sealed to a transfer line 74 leading to the sample stream inlet of the IMS sensor 10. Transfer line 74 is preferably maintained at a temperature 10°C - 40°C above (and most preferably 10°C above) that of the multicapillary column 72 in order to ensure that analyte species will not adsorb on the walls of the transfer line 72 instead of entering the sample stream inlet of the IMS sensor 10.

Figure 4, shows a preferred embodiment of the heated transfer line 74. It has a heating element 78 of one or more sections of insulated, high resistance wire, preferably 0.02 inch diameter tetraflouroethylene (TFE)-coated Constantan thermocouple wire, wound around the heated passageway of the transfer line 74 from the inlet end 75 to the outlet end 77 to provide electrical heating to the transfer line passageway 74. Other metals such as platinum that can be formed as a thin film surrounding the transfer line 74

could be used for the heating element 78. In addition, a flexible heating element 78 in which the thin metal film is embedded in a material such as a clear polyimide film, a fiber paper aramide, or silicon rubber could be used. Additionally, the thin metal film heating element 78 could be embedded in a rigid mica shell. The heating element wires 78 of the preferred embodiment is wound in a single or preferably a dual-helix fashion around the transfer line passageway 74. Preferably, the heating element wires 78 penetrate through holes in two bonded cylindrical half sections 90 of insulating material such as Kel-F plastic that is embedded between metal shells 76a and 76b. Alternately, at least one electrical contact pin made of #2-56 stainless steel all-thread rod can be mounted in one or both of the half sections 90. Metal shells 76a and 76b can be welded to transfer line 74 and are preferably supported by the cylindrical half-sections 90 and Orings (not shown) or other means known in the art. In the preferred embodiment, a layer of teflon tape 95 preferably holds the heating element wires 78 against the transfer line passageway 74 to improve heat transfer efficiency between the heating element wires 78 and the transfer line passageway 74. To provide a more uniform temperature profile across the length of the passageway 5, the winding density (or pitch) of the heating element wires 78 in the preferred embodiment is increased near the heating device inlet end 75 and outlet end 77 where heat losses are at their maximum.

Two wire resistance thermometers 91a and 91b, each preferably a Minco Thermal-Ribbon platinum resistance-measuring thermometer Model No. S651PDZ24A, are preferably attached to the transfer line passageway 74 to monitor the temperature of the transfer line 74. Other components which could be used for the thermometers 91a and 91b are a thermocouple, a thermistor, and a semiconductor. The first thermometer 91a is preferably positioned near the midpoint of the transfer line 74 while the second

thermometer 91b is preferably positioned near the outlet end 77. The thermometers 91a and 91b are preferably connected to a temperature controller which switches power to the heating element section(s) 78 to maintain a substantially uniform temperature profile across the transfer line passageway 74. A conventional resistance-measuring temperature controller, such as a MINCO Model No. CT-149, preferably compares the temperature measured by the thermometer 91a or 91b to a selected setpoint temperature programmed into the temperature controller. The temperature comparison can be performed by means of an algorithm run on a computer processor by computer software programmed for this purpose that is stored on a computer-readable storage medium. Other means of performing the temperature comparison are also possible, such as use of an electronic integrated differential amplifier circuit, an analog or a combined analog and digital comparison circuit comprised of discrete electrical and digital logic components, or other similar circuitry presently in the state of the art. As shown in Figure 7, the temperature control circuitry in the preferred embodiment results in a stabilized temperature profile across the transfer line passageway 74.

Figure 6 shows a simplified representation of the IMS sensor's response time and recovery time characteristic to a stepwise increase and decrease in the concentration of the analyte with and without heating of the IMS sensor 10 ionization chamber inlet of U.S. Patent No. 5,420,424. As can be seen from Figure 6, the IMS sensor's response exhibits a time lag from the point T1 when the concentration is increased to the point Tp when the IMS sensor's response initially reaches its peak value Cp. This time lag is due to adsorption of the analyte on the IMS sensor ionization chamber surface as the sample passes from the outside environment into the IMS sensor. This adsorption results in less of the analyte being available for ionization and detection than is actually present in the

sample. The peak IMS sensor response Cp is obtained when the concentration level of the analyte on the IMS sensor ionization chamber surface has achieved an equilibrium density that corresponds to the increased concentration level of the analyte present in the sample. At the time Tp when the equilibrium density is reached, adsorption of the analyte taking place on the IMS sensor's sample stream inlet surface is balanced by desorption of the analyte from the sample stream inlet so that no net adsorption of the analyte is occurring. This causes the full amount of the analyte entering the IMS sensor ionization chamber to be available for ionization and detection.

The increase in the analyte concentration is then eliminated at a point in time T2 very soon after the equilibrium point Tp is reached. This stepwise decrease in the original concentration level causes the reverse effect to take place and results in an additional time lag until the original analyte concentration level Co is detected by the IMS sensor at time To. In that case gradual release of the desorbed analyte from the IMS sensor ionization chamber surface results in more of the analyte being available for ionization and detection than is actually present in the sample. The original IMS sensor response Co is obtained when the concentration level of the analyte on the ionization chamber surface has returned to the equilibrium density corresponding to the original concentration level of the analyte present in the sample. The time lag in either case is directly dependent on the temperature inside the IMS sensor inlet. A greater temperature results in less time between adsorption and desorption taking place on the ionization chamber surface and a consequently smaller period of elapsed time until equilibrium is reached. The heating device shown in Figure 4 provides a means for increasing the temperature inside the IMS sensor inlet to minimize the time lag experienced in the detection of a change in analyte concentration level.

Figure 8 illustrates the effect of using a heated inlet transfer line 74 or 92 such as that shown in Figure 4 or Figure 5, respectively, on the IMS sensor's response to changes in analyte concentration levels. The use of the heated transfer line 74 or 92 to heat the IMS sensor 10 inlet to a typical operating temperature (approximately 126C) causes an approximately tenfold improvement in the IMS sensor's response time when compared to maintaining the inlet at room temperature (as indicated by the time interval from T1 to Tp' with use of the heated transfer line as opposed to the time interval from T1 to Tp without the heated transfer line installed). As can be seen from Figure 8, the IMS sensor's recovery time characteristic is also improved by use of the inlet heated transfer line (as indicated by the time interval from T2' to To with use of the heated transfer line as opposed to the time interval from T2 to a point beyond the end of the graph without the heated transfer line installed).

In order to prevent degradation of the IMS sensor's sensitivity, mixing of the carrier and sample fluid stream flows must be prevented as the carrier stream enters the IMS sensor's analytical gap from the IMS carrier stream plenum, as described in U.S. Patent No. 5,420,424. This is necessary in order to prevent impurities present in the sample fluid stream from entering the analytical gap and adversely affecting the IMS sensor ion detector measurement. To achieve the segregation of carrier and sample stream flows, the amount of flow entering the IMS carrier stream plenum typically exceeds the portion of the carrier stream flow entering the analytical gap 25 by 1 to 1.5 liters per minute. However, in order to prevent degradation of the sensitivity (i.e. change in signal amplitude per unit change in analyte concentration), the IMS carrier stream plenum flow must be on the order of 3 to 4 liters per minute. Lowering this flow level results in additional time for the ions to traverse the IMS sensor analytical gap to the ion

detector, which permits more of the ions to migrate to the analytical gap walls due to diffusion or to undergo charge neutralization reactions and thus escape measurement by the ion detector. Use of a inlet heated transfer line 74 or 92 such as that shown in Figures 4 and 5, respectively, makes it difficult to prevent adegradation in the sensitivity of the IMS sensor's response while at the same time avoiding mixing of the carrier and sample stream flows, since the maximum IMS carrier stream plenum flow possible without causing mixing is approximately 1.8 liters per minute. Mixing is caused at higher carrier stream flow levels due to the turbulence created by the blunt shape of the heating device outlet end 77 shown in Figure 4.

As shown in Figure 5 a flow smoothing insert 93 can be placed in the gap between a heated transfer line shell 76c and the inner surface of the IMS housing 11 to eliminate the turbulence created by the transfer line outlet end 77. The shape of the shell 76c reduces the gap between the IMS housing 11 inner surface and the shell 76c at the transfer line mounting flange 79 while gradually increasing this gap closer to the transfer line outlet end 77. The flow smoothing insert 93 is preferably made of a fine pitch stainless steel mesh shaped in the form of a round disc with a center opening concentric with the outer circumference of the disc. Other possible materials for the flow smoothing insert 93 are titanium, stainless steel, gold, nickel and other non-reactive metals having an inherently low vapor pressure characteristic. Other low porosity, low vapor pressure non-reactive, non-metallic materials such as glass, quartz, teflon and sapphire can be used for the flow smoothing insert 93. The flow smoothing insert 93 is preferably slipped over the transfer line outlet end 77 to form an interference fit with the transfer line shell 76c prior to mounting the heating device on the IMS sensor. When the heating device 92 is mounted on the IMS sensor 10 the flow smoothing insert 93 will fill the gap between the

transfer line shell 76c and the IMS housing 11 inner surface at a location proximate to the transfer line outlet end 77. Since the surface of the transfer line shell 76c is electrically connected (not shown) to electrode 32 to contribute to the radial electric field created by the bias potential applied to electrode 32, the flow smoothing insert 93 must be electrically insulated from the grounded IMS housing 11. This may be accomplished by placing a band 94 of electrically insulating material such as teflon between the flow smoothing insert 93 and the IMS housing 11 inner surface. After the inlet heating device 92 is mounted on the IMS sensor 10 with the flow smoothing insert 93 attached, the flow entering the carrier stream plenum 26 of the IMS sensor 10 will undergo gradual volume expansion before being filtered through the flow smoothing insert 93. This gradual volume expansion in combination with the filtering will produce a laminar carrier stream flow profile throughout the volume between the transfer line outlet end 77 and the IMS housing 11 upon exit of the carrier flow stream from the flow smoothing insert 93. This laminar flow profile prevents the sample stream from being mixed with the portion of the carrier stream entering the analytical gap of the IMS sensor 10 of U.S. Patent No. 5,420,424. This in turn will allow increasing the flow entering the carrier stream plenum 26 so that losses in IMS sensitivity due to ion diffusion in the analytical gap are minimized. Figure 9B shows the increased sensitivity of the IMS sensor's response with the flow smoothing insert 93 attached to the heated inlet transfer line 74 or 92 as compared to the IMS sensor's measurements without the flow smoothing insert 93, as shown in Figure 9A.

While presently preferred embodiments of practicing the invention has been shown and described with particularity in connection with the accompanying drawings, the invention may otherwise be embodied within the scope of the following claims.

### WHAT IS CLAIMED IS:

- 1. A fluid filtration system for use with an ion mobility spectrometer, comprising:
- A. a pump in communication with both an outlet and a carrier stream inlet of the ion mobility spectrometer for recirculating ion mobility spectrometer outlet fluid flow into the carrier stream inlet;
- B. at least one filter in communication with the pump for removing impurities from the recirculated flow; and
- C. an exhaust for removing a portion of the outlet fluid flow from the system, wherein the amount of fluid removed from the system is equal to the amount of fluid entering a sample stream inlet of the ion mobility spectrometer.
- 2. The fluid filtration system of Claim 1, wherein the ion mobility spectrometer is transportable when connected to the filtration system.
  - 3. The fluid filtration system of Claim 1, further comprising:
    - A. a first filter in communication with the inlet of the pump; and
- B. a second filter in communication with the outlet of the pump; wherein the filters prevent the migration of impurities into the ion mobility spectrometer when the filtration system is not in use.
- 4. The fluid filtration system of Claim 1, further comprising a flow restrictor in communication with the sample stream inlet of the n mobility spectrometer for controlling the amount of fluid entering the sample stream inlet.
- The fluid filtration system of Claim 1, further comprising a gas chromatograph column in communication with the sample stream inlet of the ion mobility spectrometer.

- 6. The fluid filtration system of Claim 1, further comprising:
- A. a multicapillary gas chromatograph column containing a coating comprised of a stationary phase substance in communication with the sample stream inlet for transmitting an unknown concentration level of at least one analyte into the sample stream inlet to be measured by the ion mobility spectrometer;
- B. a sample injector in communication with the multicapillary column for injecting the at least one analyte into the multicapillary column; and
- C. a source of carrier gas in communication with the sample injector for entraining the at least one analyte for transport into the multicapillary column;

wherein the at least one analyte is soluble in the stationary phase substance such that transport of the at least one analyte into the sample stream inlet will be delayed by a predetermined time interval in the multicapillary column prior to entering the sample stream inlet to be measured by the ion mobility spectrometer.

- 7. The fluid filtration system of Claim 6, wherein the stationary phase substance is selected from the group consisting of SE-30, SE-54 and carbowax 20M.
- 8. The fluid filtration system of Claim 6, wherein the sample injector contains
  a septum membrane for injecting the at least one analyte into the carrier gas.
- 9. The fluid filtration system of Claim 6, further comprising a pressure regulator in communication with the source of carrier gas for regulating the pressure of the carrier gas entering the sample injector.
- 10. The fluid filtration system of Claim 6, further comprising a heated transfer line in communication with the multicapillary column and the sample stream inlet for preventing adsorption of the at least one analyte prior to entering the sample stream inlet.

- 11. The fluid filtration system of Claim 1, further comprising a transfer line for heating the fluid stream entering the sample stream inlet of an ion mobility spectrometer, the transfer line comprising:
- A. a hollow passageway having an inlet end for receiving the fluid stream into the sample stream inlet;
- B. a hollow housing attached to both the passageway inlet and outlet ends for mounting the transfer line to the sample stream inlet of the ion mobility spectrometer;
- C. an electrical heating element surrounding the surface of the passageway for heating the passageway; and
- D. a temperature control circuit for controlling electrical power input to the heating element, comprising:
- (i) at least one temperature measuring device attached to the surface of the passageway for measuring the temperature of the passageway:
- (ii) a temperature controller having an input electrically connected to the temperature measuring device and an output electrically connected to the heating element for:
- (a) comparing the temperature measured by the temperature measuring device to a predetermined reference value; and
- (b) adjusting the electrical power input to the heating element to substantially match the temperature of the passageway to the reference value.

- 12. The fluid filtration system of Claim 5 or 6, further comprising a transfer line in communication with the gas chromatograph column for heating a fluid stream entering the sample stream inlet of an ion mobility spectrometer, the transfer line comprising:
- A. a hollow passageway having an inlet end in communication with an outlet end for passage of a sample fluid stream from the gas chromatograph column into the sample stream inlet;
- B. a hollow housing attached to both the passageway inlet and outlet ends for mounting the transfer line to the sample stream inlet of the ion mobility spectrometer;
- C. an electrical heating element surrounding the surface of the passageway for heating the passageway; and
- D. a temperature control circuit for controlling electrical power input to the heating element, comprising:
- (i) at least one temperature measuring device attached to the surface of the passageway for measuring the temperature of the passageway;
- (ii) a temperature controller having an input electrically connected to the temperature measuring device and an output electrically connected to the heating element for:
- (a) comparing the temperature measured by the temperature measuring device to a predetermined reference value; and
- (b) adjusting the electrical power input to the heating element to substantially match the temperature of the passageway to the reference value.

- 13. The transfer line of Claim 11 or 12, further comprising an element for reducing turbulence in a flow stream entering the carrier stream inlet of the ion mobility spectrometer, comprising an insert of porous material placed between the transfer line housing and the surface of the sample stream inlet at a location proximate to the outlet end of the transfer line, wherein the insert fills the gap between the transfer line housing and the sample stream inlet surface.
- 14. The apparatus of Claim 1, wherein the ion mobility spectrometer comprises:
- A. a housing having at least one inlet for communication with a sample media and at least one outlet,
  - B. an analyzer positioned within the housing comprising:
- (i) at least first and second longitudinally spaced apart electrodes, the space between the electrodes defining a longitudinal analytical gap, the gap being in communication with a source of carrier gas for flow therethrough,
- (ii) an ionization source juxtaposed with the analytical gap and in communication with the inlet for ionization of sample media,
- (iii) an ion aperture defining an opening between the ionization source and the analytical gap,
  - (iv) a third electrode positioned proximate to the ion aperture,
- (v) at least one outlet aperture from the analytical gap remote from the ion aperture,
- (vi) an ion detector for measuring ions from the analytical gap and spaced from the electrodes, and

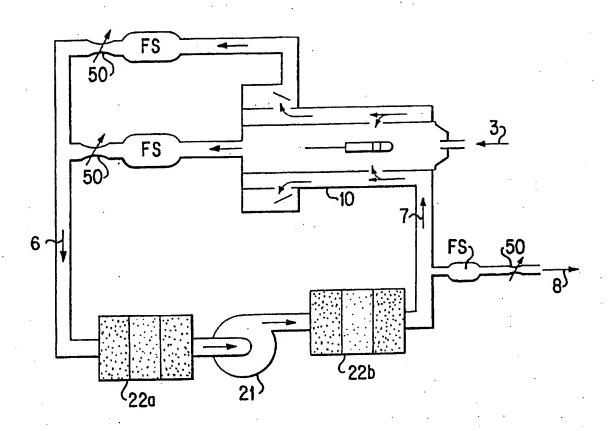
- (vii) an electrical controller connected to the electrodes for impressing:
- (a) direct current potentials to the first, second and third electrodes, and
- (b) a periodic asymmetrical potential to the first and second electrode, the potentials capable of creating a transverse electric field therebetween during the flow of carrier gas in the analytical gap.
- 15. A fluid filtration system for use with an ion mobility spectrometer in measuring an unknown concentration level of a selected ionizable substance present in a sample fluid stream in which other substances may coexist, comprising:
- A. a pump in communication with an outlet and carrier and sample stream inlets of the ion mobility spectrometer for recirculating ion mobility spectrometer outlet fluid flow into the carrier and sample stream inlets;
- B. at least one filter in communication with the pump for removing impurities from the recirculated flow;
- C. a plurality of valves in communication with the filters and the sample stream inlet for introducing the sample fluid stream into the sample stream inlet combined with first and second portions of the recirculated flow not directed to the carrier stream inlet, wherein the valves maintain a constant flow rate into the sample stream inlet;
- D. a device in communication with the valves for introducing a controlled concentration level of the selected ionizable substance into the first portion of the recirculated flow not directed to the carrier stream inlet; and

- E. an exhaust in communication with the valves for removing from the system a third portion of the recirculated flow not directed to the carrier stream inlet, wherein the amount of fluid removed equals the amount of fluid in the sample fluid stream.
- 16. The fluid filtration system of Claim 15, wherein the device in communication with the valves for introducing a controlled concentration level of the selected ionizable substance into the first portion of the recirculated flow not directed to the carrier stream inlet is selected from the group consisting of a permeation calibration device, a diffusion vial and a pressurized calibration cylinder.
- 17. A method of using the fluid filtration system of Claim 15 or 16 with an ion mobility spectrometer to measure an unknown concentration level of a selected ionizable substance present in a sample fluid stream in which other substances may coexist, comprising the following steps:
- A. introducing a controlled concentration level of the selected ionizable substance into the first portion of the recirculated flow not directed to the carrier stream inlet;
- B. combining the sample fluid stream with both the first and second portions of the recirculated flow not directed to the carrier stream inlet to measure the combined unknown and controlled concentration level;
- C. combining the sample fluid stream with the second portion of the recirculated flow not directed to the carrier stream inlet to measure the unknown concentration level;
- D. obtaining the unknown concentration by comparing the combined concentration measurement to the unknown concentration measurement.

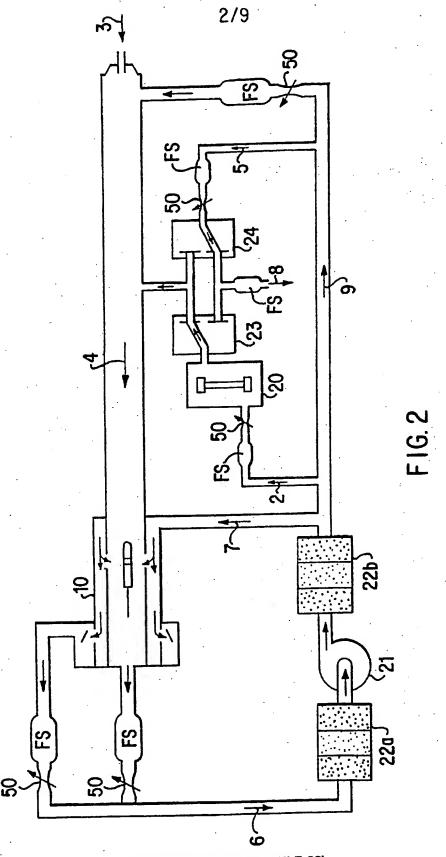
- 18. The method of Claim 17, wherein the unknown concentration level is obtained by calculating the ratio of the combined concentration measurement to the unknown concentration measurement in accordance with the Standard Addition Method.
- 19. The method of Claim 17, wherein the unknown concentration level is calculated by a device selected from the group consisting of computer processor, an electronic integrated differential amplifier circuit, an analog comparison circuit comprised of discrete electrical components, and a combined analog and digital comparison circuit comprised of discrete electrical and digital logic components.
- 20. A method of using the fluid filtration system of Claim 15 or 16 to calibrate an ion mobility spectrometer by combining the first and second portions of the recirculated flow not directed to the carrier stream inlet to measure the controlled concentration level.
  - 21. The fluid filtration system of Claim 15, further comprising:
  - A. a first filter in communication with the inlet of the pump; and
- B. a second filter in communication with the outlet of the pump; wherein the filters prevent the migration of impurities into the ion mobility spectrometer when the filtration system is not in use.
- 22. The fluid filtration system of Claim 15, wherein the ion mobility spectrometer comprises:
- A. a housing having at least one inlet for communication with a sample media and at least one outlet,
  - B. an analyzer positioned within the housing comprising:

- (i) at least first and second longitudinally spaced apart electrodes, the space between the electrodes defining a longitudinal analytical gap, the gap being in communication with a source of carrier gas for flow therethrough,
- (ii) an ionization source juxtaposed with the analytical gap and in communication with the inlet for ionization of sample media,
- (iii) an ion aperture defining an opening between the ionization source and the analytical gap,
  - (iv) a third electrode positioned proximate to the ion aperture,
- (v) at least one outlet aperture from the analytical gap remote from the ion aperture,
- (vi) an ion detector for measuring ions from the analytical gap and spaced from the electrodes, and
- (vii) an electrical controller connected to the electrodes for impressing:
- (a) direct current potentials to the first, second and third electrodes, and
- (b) a periodic asymmetrical potential to the first and second electrode, the potentials capable of creating a transverse electric field therebetween during the flow of carrier gas in the analytical gap.

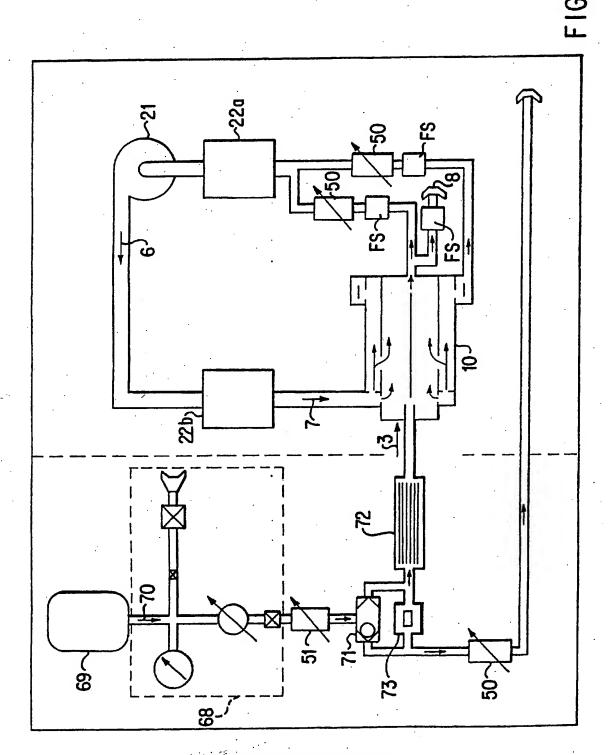
1/9



F I G. 1



SUBSTITUTE SHEET (RULE 26)



SUBSTITUTE SHEET (RULE 26)

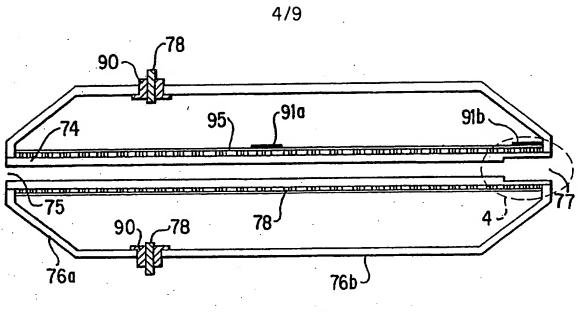
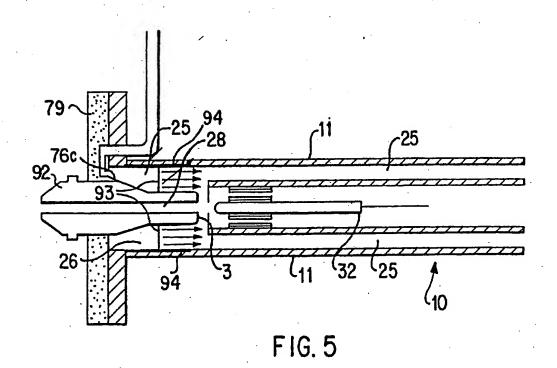
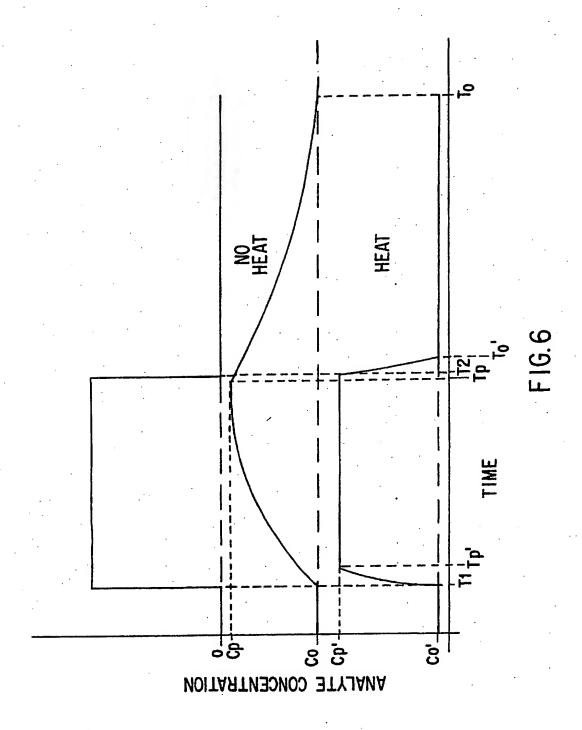


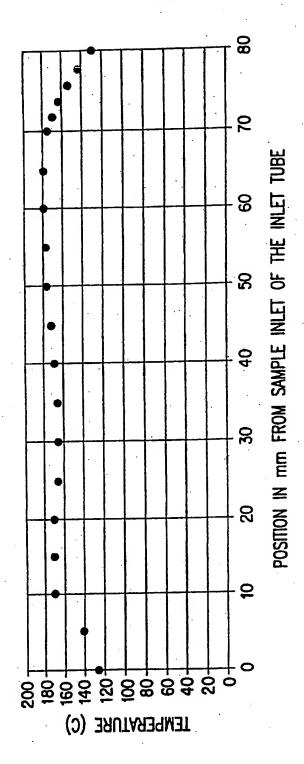
FIG. 4



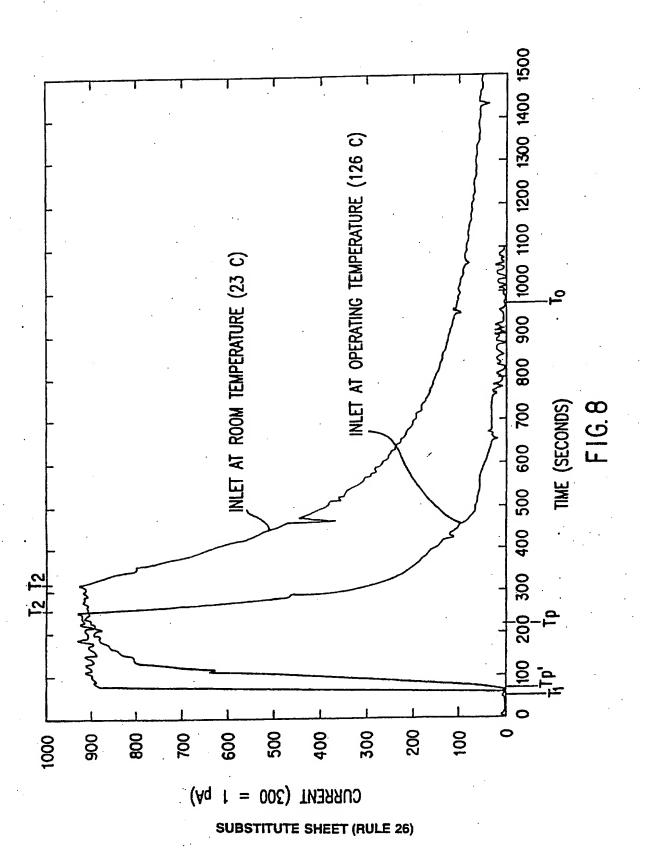
SUBSTITUTE SHEET (RULE 26)

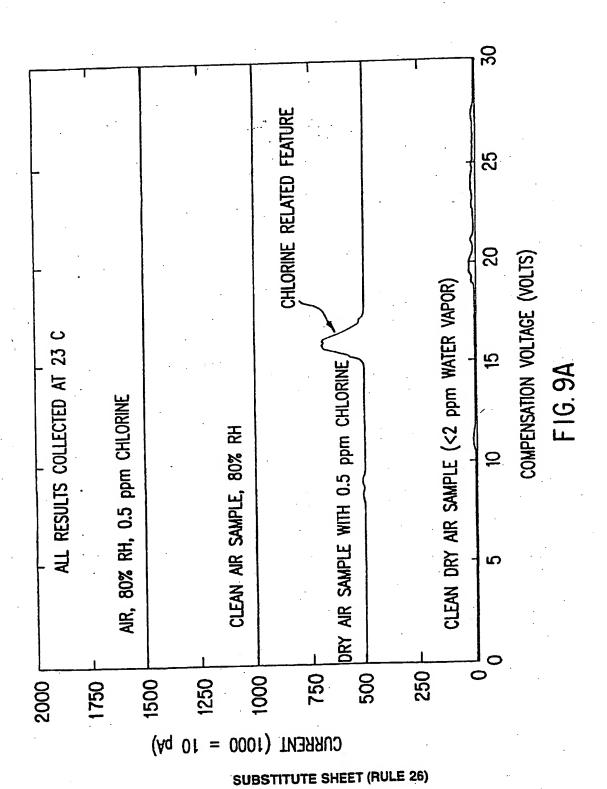


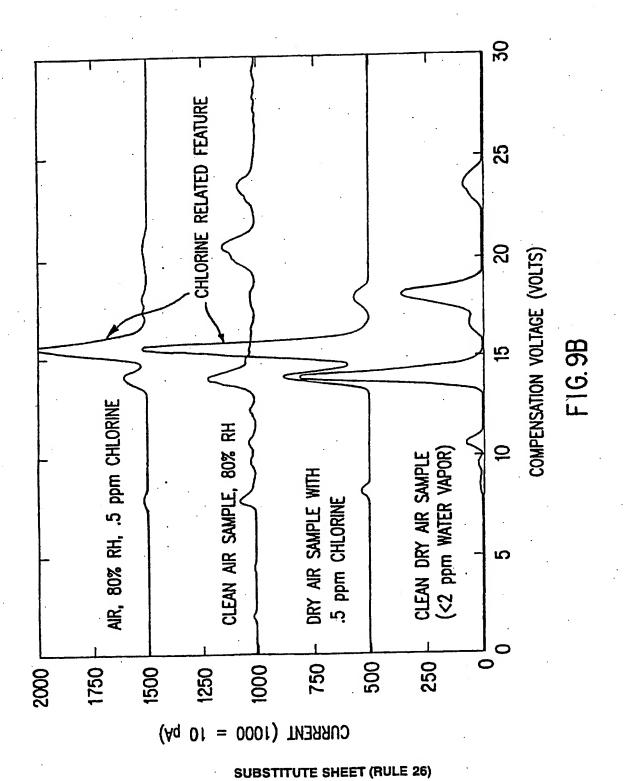
SUBSTITUTE SHEET (RULE 26)



F16.7







## INTERNATIONAL SEARCH REPORT

Intert. al Application No PCT/US 97/05641

A. JASSI	FICATION OF SUBJECT MATTER GO1N27/64	
According to	International Patent Classification (IPC) or to both national classification and IPC	
	FOA POWED	
Minimum d	ocumentation searched (classification system followed by classification symbols)  GOIN	
•		
	on searched other than minimum documentation to the extent that such documents are included in the fiel	ds searched
Documenta	IDII SEALEMA ORIAL OTT.	
•		
Electronic	iata base consulted during the international search (name of data base and, where practical, search terms us	
C. DOCU	MENTS CONSIDERED TO BE RELEVANT	Relevant to claim No.
Category *	Clauson of document, with indication, where appropriate, of the relevant passages	
<b></b>	US 4 317 995 A (R. F. D. BRADSHAW) 2 March	1
Υ	1 1000	
l	see column 3, line 14 - line 48; figure 1	
		1
Y	EP 0 135 747 A (THE BENDIX CORP.) 3 April 1985	
	see figure 4	
		1
A	ANALYTICAL CHEMISTRY, vol. 65, no. 3, 1993, COLUMBUS US,	•
	1 nage 299-386 XP00035004b	
	I A D SNYDED. "PORTABLE HAND-HELD GAS	
	CHROMATOGRAPHY/ION MOBILITY SPECTROMETRY	*
	DEVICE" see figure 2	
	-/	
1		
1		
IXI F	urther documents are listed in the continuation of box C.	listed in annex.
	for the designation of the second sec	the international filing date
	categories of cited documents The later document punished and or priority date and not in con- cument defining the general state of the art which is not cited to understand the principles.	
201	indered to be of particular relevance invention	er the claimed invention
l 65i	ng date cannot be considered novel or involve an inventive step when	the document is taken alone
	ument which may drow doubts on priority changes are in the publication date of another y document of particular relevant to the publication date of another y document of particular relevant to the publication of the publication date of another y document of particular relevant to the publication of the publication o	ce; the claimed invention
.O. 40	tument referring to an oral disclosure, use, exhibition of document is combination being ments, such combination being	
ot do	ner means in the art.  The international filing date but the international filing date but the art.	
lat	er than the priority date claimed	
Date of	the actual completion of the international search	13.08.97
	5 August 1997	12100121
.	Authorized officer	,
Name	and mailing address of the ISA  European Patent Office, P.B. 5818 Patentiaan 2	
	NL . 2280 HV Risswik Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+ 31-70) 340-3016  Duchatellier,	M

## INTERNATIONAL SEARCH REPORT

Intern al Application No PCT/US 97/05641

A US 4 259 573 A (J. M. PROBER) 31 March see abstract; figure 1  A US 5 832 721 A (A.T. BACON) 16 July 1991 see abstract; figure 1	· .	PCT/US 97/05041
A US 4 259 573 A (J. M. PROBER) 31 March 1981 see abstract; figure 1	(4 minuation) DOCUMENTS CONSIDERED TO BE RELEVANT	Rejevant to claim No.
A US 4 259 573 A (J. M. PROBER) 31 March 1981 see abstract; figure 1		
A US 5 032 721 A (A.T. BACON) 15 July 153 see abstract; figure 1	1981 see abstract; figure 1	
	A US 5 032 721 A (A.T. BACON) 16 July 1991 see abstract; figure 1	
		*
		-

## INTERNATIONAL SEARCH REPORT

Information on patent family members

Intern. at Application No PCT/US 97/05641

Patent document	Publication date	Patent family member(s)	date
US 4317995 A	02-03-82	GB 2052750 A AU 5942180 A BR 8003786 A EP 0021518 A JP 56006153 A	28-01-81 08-01-81 13-01-81 07-01-81 22-01-81
EP 135747 A	03-04-85	US 4551624 A DE 3485754 A	05-11-85 09-07-92
US 4259573 A	31-03-81	NONE	
US 5032721 A	16-07-91	US 5095206 A US 5234838 A US 5283199 A	10-03-92 10-08-93 01-02-94